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REPORT OF INVESTIGATIONS/1989

Determining the Relative Toxicity and Smoke Obscuration of Combustion Products of Mine Combustibles

By Maria I. De Rosa and Charles D. Litton

BUREAU OF MINES

UNITED STATES DEPARTMENT OF THE INTERIOR



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**UNITED STATES DEPARTMENT OF THE INTERIOR
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CONTENTS

	<i>Page</i>
Abstract	1
Introduction	2
Principles of operation of submicrometer particle detector	2
Calibration	4
Experimental system	4
Experimental procedure	5
Results and discussion	5
Smoke obscuration	5
Toxicity	7
Conclusions	9
References	9
Appendix.—Symbols	10

ILLUSTRATIONS

1. Ionization chamber used to measure particle diameter and concentration	2
2. Particle size calibration data for submicrometer particle detection	4
3. Experimental setup used to measure smoke characteristics	4
4. Correlation of relative optical densities ($d_g^2 n_o$) with parameter $d_g n_o / \Delta m$	6
5. Comparison of average particle diameter (d_g) for a jute brattice at different furnace temperatures	7
6. Comparison of value of $d_g n_o$ for a jute brattice at different furnace temperatures	7
7. Correlation of parameter $1/d_g n_o$ with toxic load data obtained by other researchers	8

TABLE

1. Experimental data related to smoke optical density	6
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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

A	ampere	L/min	liter per minute
C	coulomb	m	meter
°C	degree Celsius	min	minute
cm	centimeter	μCi	microcurie
cm ³	cubic centimeter	μm	micrometer
cm ² /p	square centimeter per particle	p/cm	particle per centimeter
cm/s	centimeter per second	p/cm ²	particle per square centimeter
cm ² /s	square centimeter per second	p/cm ³	particle per cubic centimeter
cm ³ /s	cubic centimeter per second	p/(cm ² · g)	particle per square centimeter per gram
cm ² /V-s	square centimeter per volt-second	pct	percent
esu	electrostatic unit	s	second
g	gram	V	volt
L	liter		

DETERMINING THE RELATIVE TOXICITY AND SMOKE OBSCURATION OF COMBUSTION PRODUCTS OF MINE COMBUSTIBLES

By Maria I. De Rosa¹ and Charles D. Litton²

ABSTRACT

Combustible materials, when burned, produce toxic gases and smoke, which may vary dramatically from one material to another, with resultant different total toxicity and smoke obscuration levels. This U.S. Bureau of Mines report presents smoke property data acquired for a variety of mine combustibles and shows that there exists a correlation between the smoke properties, the relative toxicity, and the smoke obscuration levels of those combustibles tested. These correlations can be used to devise simple, standard tests for determining the toxic and smoke obscuration hazards of mine materials during fire.

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INTRODUCTION

During combustion, materials generate smoke, toxic gases, and soot particles, which during a fire, severely reduce the potential for escape; it is well known that the majority of fire deaths are due to the effects of these products rather than to the heat from the fire (1).³ Not surprisingly, the toxicities of gases and densities of smoke evolving from the combustion of various materials have been the subject of intense research by the fire research community. Smoke, by hindering visibility, slows down escape efforts, increasing the exposure time to the toxic gases and to the toxic, respirable soot particles evolved during the fire. It is, then, the combination of gas toxicity, smoke obscuration, and their synergistic effects that forms the total hazard to life safety.

Relatively simple tests have been developed for determining the smoke levels produced from combustible materials. A good example of this type of test is the National Bureau of Standards (now the National Institute of Standards and Technology) smoke chamber method (2). Other, similar tests also exist. However, tests to determine the toxic hazard of smoke are generally elaborate and time consuming. The analytical approach requires the precise measurement of many toxic gases, while the experimental approach utilizes the responses of laboratory animals to the toxic gases in the smoke to derive the relative toxicities of different combustible materials.

Most importantly, no single test exists that measures both the smoke obscuration hazard and the toxic hazard in a simple, straightforward fashion. If such a test could be devised, what form would it take? What parameters should be measured?

In 1984, the U.S. Bureau of Mines began to study the physical properties of smoke produced from different combustible mine materials during oxidative thermal degradation, utilizing a well-defined ionization chamber to obtain real-time average diameters and concentrations of the smoke particles. From the outset of these experiments, it was hoped that some property, or combination of properties, of the different experimental smokes could be related to the relative gas toxicities of the combustibles, as well as providing information on the relative smoke obscurations that could result.

This report describes the tests that were conducted and the results that were obtained. It is the intent of this report to demonstrate that there exists a single parameter of the smoke that can be used reliably to determine both the toxicity of one combustible material relative to another and the relative smoke obscuration hazards of different combustibles.

PRINCIPLES OF OPERATION OF SUBMICROMETER PARTICLE DETECTOR

The submicrometer particle detector (SMPD) used in the current studies for the measurement of smoke properties has been described in detail elsewhere (3); for these studies it was modified so that smoke particle mean diameter (d_p), in centimeters, and smoke particle concentration (n_p), in particles per cubic centimeter, could be obtained in real time. These modifications and the principles of operation of the device are described below. A schematic of a typical ionization chamber used in the SMPD is shown in figure 1.

The SMPD operates via the principle of diffusion charging of smoke particles in a unipolar ion region that is created in an air space between two cylindrically concentric electrodes. The center, source electrode is americium-241 cast in the shape of a right circular cylinder of 0.25 cm radius and has a uniform activity across its surface. The source electrode has a total activity of 150 μ Ci and is covered with a gold foil $\sim 7.0 \mu$ m thick to limit the range of penetration of emitted alpha particles to an average value of 0.75 cm from the source surface. The outer electrode is fixed at a radial distance from the center of 1.588 cm (1.34 cm from the source surface). When a

positive voltage is applied to the source electrode and the outer electrode voltage is held at zero (ground), positive ions move out of the region of primary ionization ($r \leq 1.00$ cm) toward the outer electrode, while negative ions are swept to the center, source electrode. This produces a region of unipolar (positive) ions in the air space defined by $1.0 \text{ cm} < r < 1.588 \text{ cm}$. Because the electric field also acts to separate the positive and negative ions within the region of primary ionization, an "effective" region of unipolar positive ions actually exists that accounts for over 95 pct of the total volume between the electrodes.

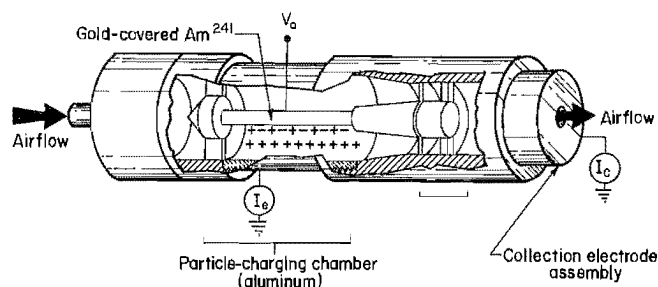


Figure 1.—Ionization chamber used to measure particle diameter and concentration.

³Italic numbers in parentheses refer to items in the list of references preceding the appendix at the end of this report.

A small, internal pump continuously pulls air at a constant flow rate through the ionization chamber. Smoke particles entrained in the flow acquire some net positive charge as they flow through this region. The total charge acquired by an individual particle depends upon the diameter of the particle (d_p), the unipolar ion concentration (n^+), and the residence time (t) of the particle within the chamber. The unipolar ion concentration per cubic centimeter is calculated from the measured current (I_e) at the outer electrode from the expression

$$n^+ = (I_e/V_o)[\ln(R_o/r_o)/(2\pi e' \mu^+ l_o)], \quad (1)$$

where V_o = voltage between the two electrodes, 71.3 V,

R_o = outer electrode radius, 1.588 cm,

r_o = source electrode radius, 0.25 cm,

l_o = length of chamber, 5.9 cm,

e' = electronic charge (1.6×10^{-19} C),

and μ^+ = positive ion electrical mobility, $1.4 \text{ cm}^2/\text{V-s}$.

The residence time (t), in seconds, is calculated by dividing the chamber volume by the flow rate (Q_o), in cubic centimeters per second, or

$$t = [\pi(R_o^2 - r_o^2)l_o]/Q_o = 46.12/Q_o. \quad (2)$$

Combining equations 1 and 2, the product n^+t is defined by

$$n^+t = 1.42 \times 10^{17} (I_e/Q_o). \quad (3)$$

In the SMPD chamber, the current (I_e) is amplified and converted to a voltage (V_{oe}) via the expression

$$V_{oe} = 2.388 \times 10^8 I_e,$$

so that

$$n^+t = 5.95 \times 10^8 (V_{oe}/Q_o). \quad (4)$$

The average charge that any individual particle acquires is determined from the expression

$$N_p = (d_p kT/2e^2) n [1 + (\pi e^2 \bar{c} d_p n^+ t/2kT)], \quad (5)$$

where k = Boltzmann constant, 1.38×10^{-16} erg per kelvin,

T = temperature, 295 kelvins,

e = electronic charge, 4.8×10^{-10} esu,

and \bar{c} = root mean thermal ion speed, 2.4×10^4 cm/s.

It is convenient to define a charging parameter (γ), by

$$\gamma = (\pi e^2 \bar{c} n^+ t/2kT) = 1.27 \times 10^8 (V_{oe}/Q_o), \quad (6)$$

which, for the constant values of $V_{oe} = 1.03$ V and $Q_o = 27.0 \text{ cm}^3/\text{s}$ used in these studies, becomes

$$\gamma = 4.845 \times 10^6,$$

and N_p reduces to

$$N_p = 8.835 \times 10^4 d_p \ln(1 + 4.845 \times 10^6 d_p). \quad (7)$$

It can be shown that for a system of polydisperse smoke particles of mean diameter d_p , the average charge acquired by those particles can be expressed by equation 7 using the value of $d_p = d_g$.

Once the particles are charged and flow out of the charging region, they are collected at a third electrode, producing a current (I_c), which is amplified and converted to a voltage (V_c), according to

$$V_c = 2.5 \times 10^{10} I_c = 2.5 \times 10^{10} e' \bar{N}_p n_o Q_o, \quad (8)$$

where \bar{N}_p represents the average number of charges acquired by a system of polydisperse particles of mean diameter d_g and is given by equation 8 with $d_p = d_g$. Combining equations 7 and 8 yields

$$V_c = 9.542 \times 10^{-3} d_g n_o \ln(1 + 4.845 \times 10^6 d_g). \quad (9)$$

This expression may be solved for d_g in terms of V_c and the product $d_g n_o$, yielding

$$d_g = 2.064 \times 10^{-7} (\exp(104.8 V_c/d_g n_o) - 1). \quad (10)$$

V_c is measured for each experiment on a continuous basis; only the product $d_g n_o$ needs to be determined in order to determine d_g . This product can be determined by measuring the voltage reduction at the outer electrode of the ionization chamber that occurs when smoke particles are present. If the ratio η is defined by

$$\eta = V_{oe}(d_g n_o)/(V_{oe})_o,$$

where $V_{oe}(d_g n_o)$ is the voltage that exists in the presence of smoke particles and $(V_{oe})_o$ is the voltage that exists when no smoke is present, the parameter η may be defined by

$$\eta = (1/K d_g n_o) [1 - \exp(-K d_g n_o)], \quad (11)$$

where K is the effective cross section for ionization for a particular chamber and may be defined theoretically by the expression (4)

$$K = 2\pi D_i (R_o^2 - r_o^2) \ln(R_o/r_o) / (\mu^+ V_o). \quad (12)$$

D_i is the positive ion diffusion coefficient with a value of $0.036 \text{ cm}^2/\text{s}$. The theoretical value of K is calculated to be $K = 0.012 \text{ cm}^2/\text{p}$.

CALIBRATION

Once K is known, any measured value of η may be used to determine $d_g n_o$ from equation 11. This value, along with the measured value of V_c , can then be used to determine d_g from equation 10. To determine the validity of equations 10 and 11, the SMPD was calibrated using aerosol particles of known average diameter, produced from a commercially available monodisperse aerosol generator. For each particle size, three measurements were made at three different concentration levels. A total of 14 particle sizes were measured, spanning the range from $d_g = 0.035 \mu\text{m}$ to $d_g = 0.886 \mu\text{m}$. The acquired data for each test, namely, η and V_c , were then used to calculate the number mean diameter (d_g) from equations 10 and 11, using the theoretical value of K . The results are displayed in

figure 2, where the ordinate represents the measured value using the η and V_c data and the abscissa represents the value expected from the aerosol generator. There is nearly perfect agreement between the measured and expected values. The data point circles represent the average of the three measurements for each particle size and concentration. The maximum deviation from the average for any set of three measurements was less than 15 pct, while the mean deviation from the average was less than 8 pct.

It was with some degree of confidence, then, that a series of tests were undertaken to determine, in real time, the values of d_g and n_o that were produced from the oxidative thermal degradation of combustible materials.

EXPERIMENTAL SYSTEM

The experimental setup used in these studies is shown in figure 3 and consists of the following major components:

1. The combustion furnace has an operating temperature range of 373 to 1,473 kelvins.
2. A sensitive load cell is connected through the furnace floor via a quartz pedestal to a sample cup. The output of the load cell is amplified and continuously recorded on a strip-chart recorder.
3. Thermocouples monitor both furnace temperature and sample temperature.

4. A small hole in the center of the furnace door is used to continuously ventilate the furnace during a typical test. The flow rate is maintained at 10 L/min, which, for a furnace volume of 18.6 L, results in a complete change of air every 1.86 min.

5. A small diaphragm pump with internal components coated with fluorocarbon polymer is used to provide the ventilating airflow.

6. The effluent from this pump is connected via a T connector to the ionization chamber of the SMPD previously described. Flow through the ionization chamber is held constant at $27.0 \text{ cm}^3/\text{s}$. The remainder of the flow is exhausted through a laboratory fume hood to outside air.

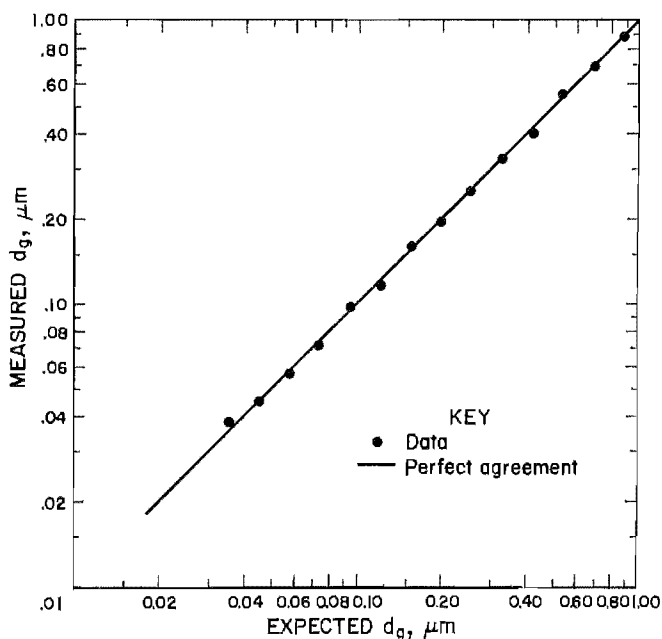


Figure 2.—Particle size calibration data for submicrometer particle detection.

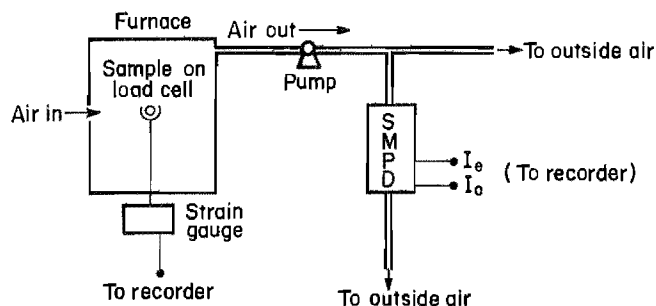


Figure 3.—Experimental setup used to measure smoke characteristics.

EXPERIMENTAL PROCEDURE

For each material tested, a 2-g sample was placed in the sample cup and positioned in the center of the oven on the quartz pedestal. The furnace door was then closed and the pump turned on to provide the ventilating airflow. The furnace temperature was selected and set, initiating the test at time $t = 0$.

Smoke produced during each test was monitored continuously with the ionization chamber outputs, V_{oe} and V_c , as previously discussed. Following an initial set of exploratory tests at various furnace temperatures, it was decided to standardize the temperature at 250° C. At this preset temperature, the furnace actually reached a peak level of 330° C at the 9th minute; the temperature then declined to a stable value of 250° C between the 10th and 11th minutes. It was also decided to standardize the time period over which data were collected to 10 min, beginning at minute 4 and ending at minute 14. The rationale for this was the observation that the combustible samples did not begin to thermally decompose until after minute 4, and at

the other extreme, significant charring was not evident until after minute 14. The 10-min test period always contained the period of most rapid devolatilization and the onset of charring.

There were two series of tests. In the first series, V_c and V_{oe} data during the test period were continuously recorded, and average values of d_g and $d_g n_o$ were obtained from the recorder traces. These tests were used to determine a smoke obscuration parameter as discussed below.

In the second series of tests, a time-averaged value of V_{oe} was obtained using an electronic integrating circuit from which the time-averaged value of $d_g n_o$ could be obtained. These data were subsequently used to derive a toxicity parameter.

In retrospect, both V_c and V_{oe} should be integrated during the 10-min test to derive average values of d_g and $d_g n_o$. This is currently being done in preparation for subsequent tests.

RESULTS AND DISCUSSION

The data acquired from these tests are divided into two sections, the first dealing with the correlations derived for smoke obscuration, and the second with the correlations derived for gas toxicity.

SMOKE OBSCURATION

The conventional tests for relative smoke obscuration levels use a light source and a detector that simulates the visual wavelength response of the human eye at some fixed separation (l). Smoke particles attenuate the light, according to Beer's law, and an optical density (σ_l) for the material may be measured. Beer's law states that the transmission of light through a cloud of smoke is given by

$$T\ell = e^{-\sigma_l}$$

and the optical density is then defined by

$$\sigma_l = (1/l) \ln (1/T\ell),$$

for a polydisperse system of smoke particles,

$$\sigma_l = \bar{K} \pi d_g^2 n_o / 4,$$

where \bar{K} is the integrated value of the Mie extinction coefficient over the particle size distribution of the smoke particles.

Now, \bar{K} also depends upon the refractive index of the particles, which means that it depends to a certain degree upon the chemistry of the particles. For distributions of small particle diameters ($d_g < 0.20 \mu\text{m}$), absorbing particles

attenuate light much more efficiently than nonabsorbing particles. For $d_g > 0.20 \mu\text{m}$, there is little difference in \bar{K} , and σ_l then depends primarily upon $d_g^2 n_o$.

Table 1 lists the experimental average values of $d_g n_o$, d_g , and Δm (the sample mass loss) for seven combustible materials tested with the ionization chamber. All of the materials tested had values of $d_g > 0.20 \mu\text{m}$, although the data for wood ($d_g = 0.22 \mu\text{m}$) are marginal. Consequently, for these data, relative values of $d_g^2 n_o$ would yield relative values of the optical densities. Column 5 gives the calculated value of a parameter, $d_g n_o / \Delta m$, which is termed the "relative smoke obscuration hazard" (RSH).

Figure 4 is a plot of $d_g^2 n_o$ versus $d_g n_o / \Delta m$. As can be seen, the data scale very nicely according to the equation

$$d_g^2 n_o = 3.82 \times 10^{-8} (d_g n_o / \Delta m)^{0.508}.$$

The values of d_g and $d_g n_o$ are the average values obtained over a constant period of time (600 s or 10 min). Within this time interval, which begins at minute 4 and ends at minute 14, the combustibles appear to go through about three stages. In the first stage, the material is beginning to heat, producing low concentrations of particles of relatively large average diameter. In the second stage, the material undergoes rapid devolatilization, producing high concentrations of particles of much smaller average diameter. And in the third stage, the onset of char formation occurs, in which the concentrations gradually decline and the average particle diameter increases. Typical plots of d_g and $d_g n_o$ versus time for a burlap brattice are shown in figures 5 and 6, illustrating these stages. The use of average values, then, represents some time-averaged value of the optical density as the combustible undergoes the transition from virgin material to eventual char.

Given that it is possible to utilize the ionization chamber to determine relative values of smoke obscuration through the use of the smoke obscuration hazard

parameter $d_g n_o / \Delta m$, what statements, if any, can be made concerning the relative toxicities of the different combustibles utilizing the ionization chamber?

Table 1.—Experimental data related to smoke optical density

Material	$d_g n_o$, p/cm^2	d_g , μm	Δm , g	$d_g^2 n_o$, p/cm	$d_g n_o / \Delta m$, $p/(cm^2 \cdot g)$
Polyvinyl chloride (PVC) brattice	37.5	0.67	1.25	25.1	30.0
Pittsburgh Seam coal	27.5	1.02	.49	28.1	56.1
Burlap brattice	110	.31	1.40	34.1	78.6
Wood	159	.22	1.60	35.0	99.4
Electrical Insulation	115	.63	.30	72.5	383.3
65 pct cotton, 35 pct polyester	104	.96	.20	99.8	520.0
Rubber mat	170	.80	.167	136.0	1,108.0

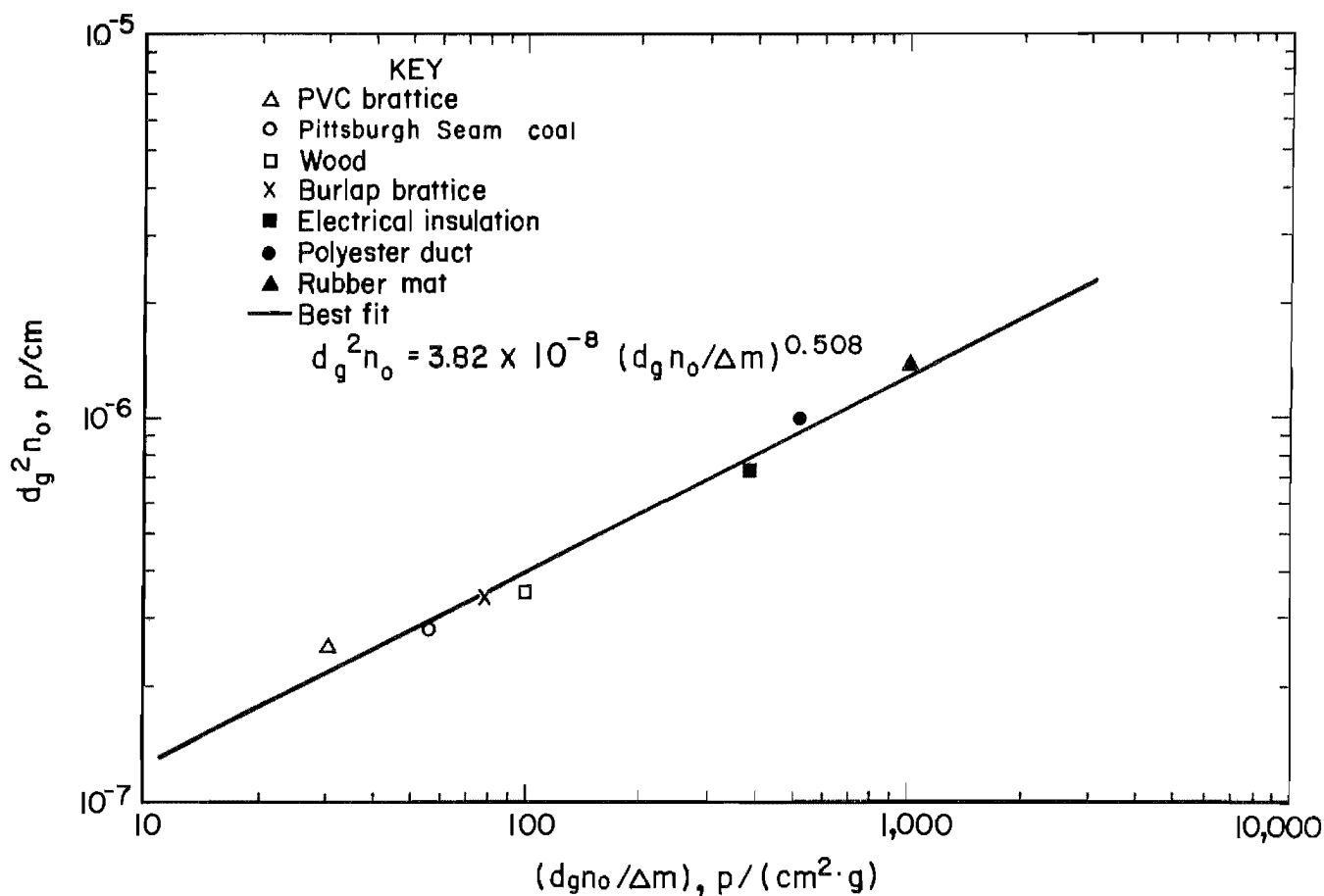


Figure 4.—Correlation of relative optical densities ($d_g^2 n_o$) with parameter $d_g n_o / \Delta m$.

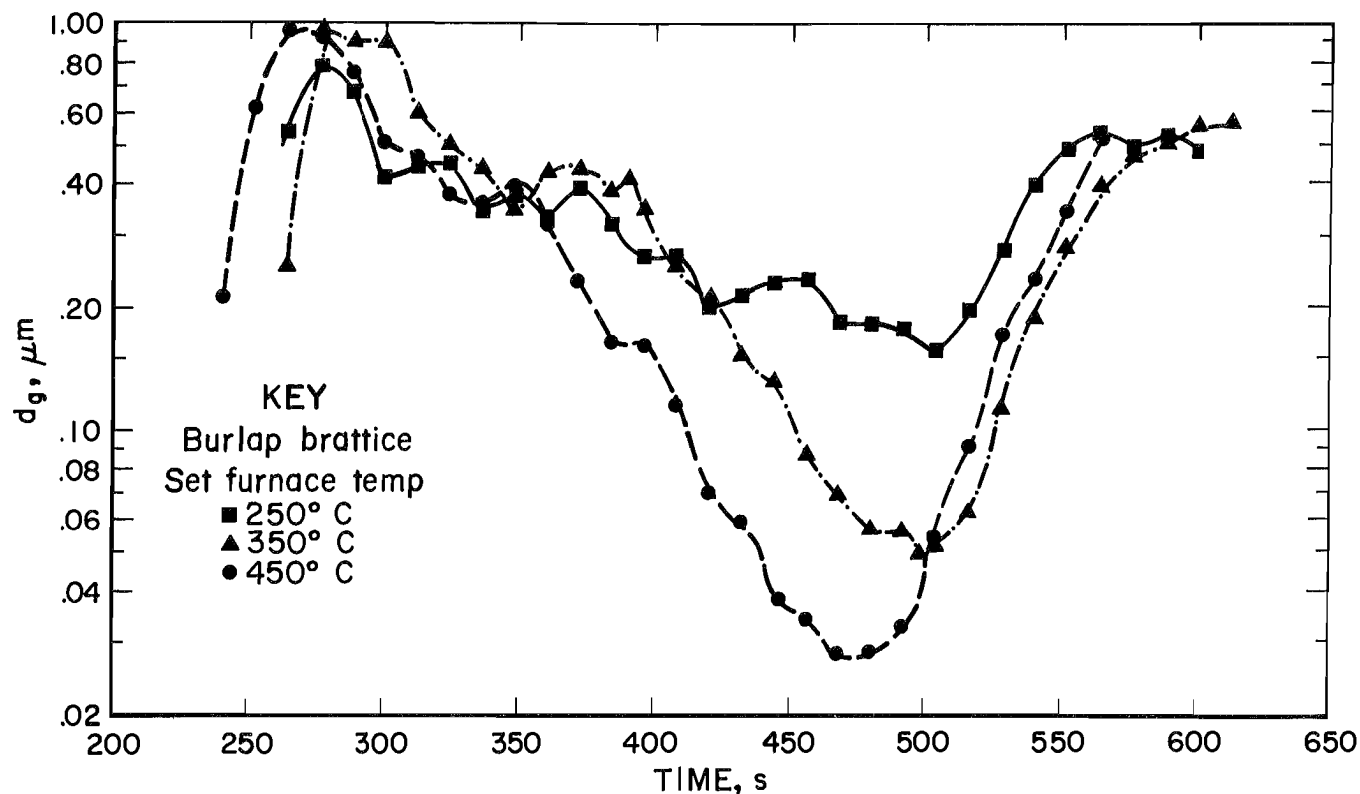


Figure 5.—Comparison of average particle diameter (d_g) for a jute brattice at different furnace temperatures.

TOXICITY

Toxicity data are usually derived in one of two ways. The first method is the analytical, in which measurements of the toxic gases generated are utilized to determine the total toxic load produced by a combustible. The second method is the experimental, in which laboratory animals are exposed to the toxic combustion products, and the quantity of combustible necessary to produce a 50-pct mortality rate (LC50) or the time of exposure necessary to produce a 50-pct mortality rate (LT50) is measured for each combustible.

To provide a basis for comparing the data obtained in this study with existing toxicity data, two previously published reports were selected. In the first report, by Ultra-systems, Inc. (5), the yields of toxic gases were determined for each combustible sample, in terms of the mass of a toxic gas produced per mass of sample tested. Each toxic gas yield (Y_i) is divided by the threshold limit value (TLV_i) for that gas, and the sum of $Y_i/(TLV_i)$ for all toxic gases is expressed as the total toxic load for that combustible. That is,

$$\text{Toxic load} = \sum_{i=1}^n Y_i / (TLV_i).$$

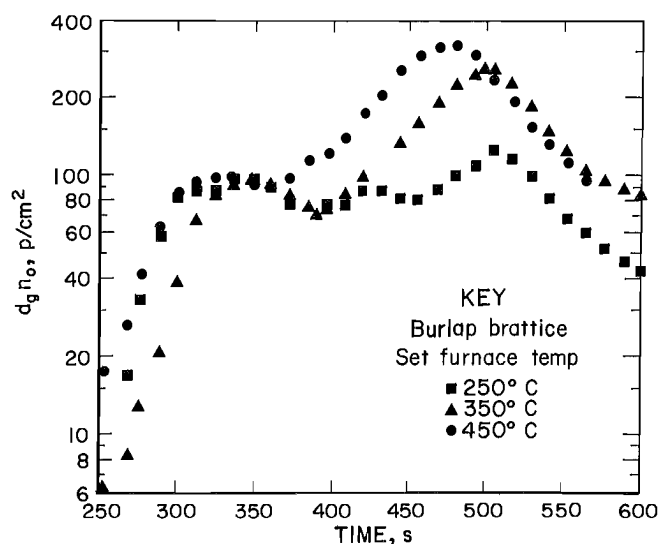


Figure 6.—Comparison of value of $d_g n_o$ for a jute brattice at different furnace temperatures.

In the second report, by Alarie and Anderson (6), the LT50 and LC50 values were measured and combined to produce an LTC50 value. To equate the data from these independent tests, the average LTC50 value for wood reported by Alarie and Anderson was normalized to the average toxic load (TL) for wood reported by Ultra-systems, Inc., according to

$$C_o / (LTC50)_{\text{wood}} = (TL)_{\text{wood}}.$$

For wood, C_o had a value of 4,600. All other LTC50 values were scaled to toxic load according to

$$TL = 4,600 / (LTC50).$$

The toxic loads derived from the Alarie and Anderson data, via the above relationship, were in excellent agreement with the toxic loads derived analytically by Ultra-systems, Inc.

For the data acquired in the series of tests reported here, the time-averaged value of the product $d_g n_o$ was obtained for each material for a constant time interval of 600 s, starting at $t = 4$ min and ending at $t = 14$ min. The

inverse of this average value ($1/d_g n_o$), is plotted in figure 7 as a function of the toxic load or relative toxicity hazard (RTH) obtained from the two cited reports. For each combustible material, the symbols represent the average of the data for $1/d_g n_o$ at the average value of the toxic load obtained from the two reference reports. The rectangles around each symbol correspond, along the abscissa, to the total range of toxic loads for both reports, and along the ordinate, to the range of values of $1/d_g n_o$ obtained in these series of tests for each type of combustible.

From figure 7, it is apparent that the parameter $1/d_g n_o$ correlates very well with the relative toxicity of the various combustibles. A simple power curve fit to the data is defined by the solid line of figure 7 and is given by the expression

$$RTH = 2.303 \times 10^5 (1/d_g n_o)^{2.192}.$$

The units of $1/d_g n_o$ are square centimeter per particle and represent some measure of the effective cross section for ionization of the different experimental "smokes." This cross section also scales with the relative toxicity.

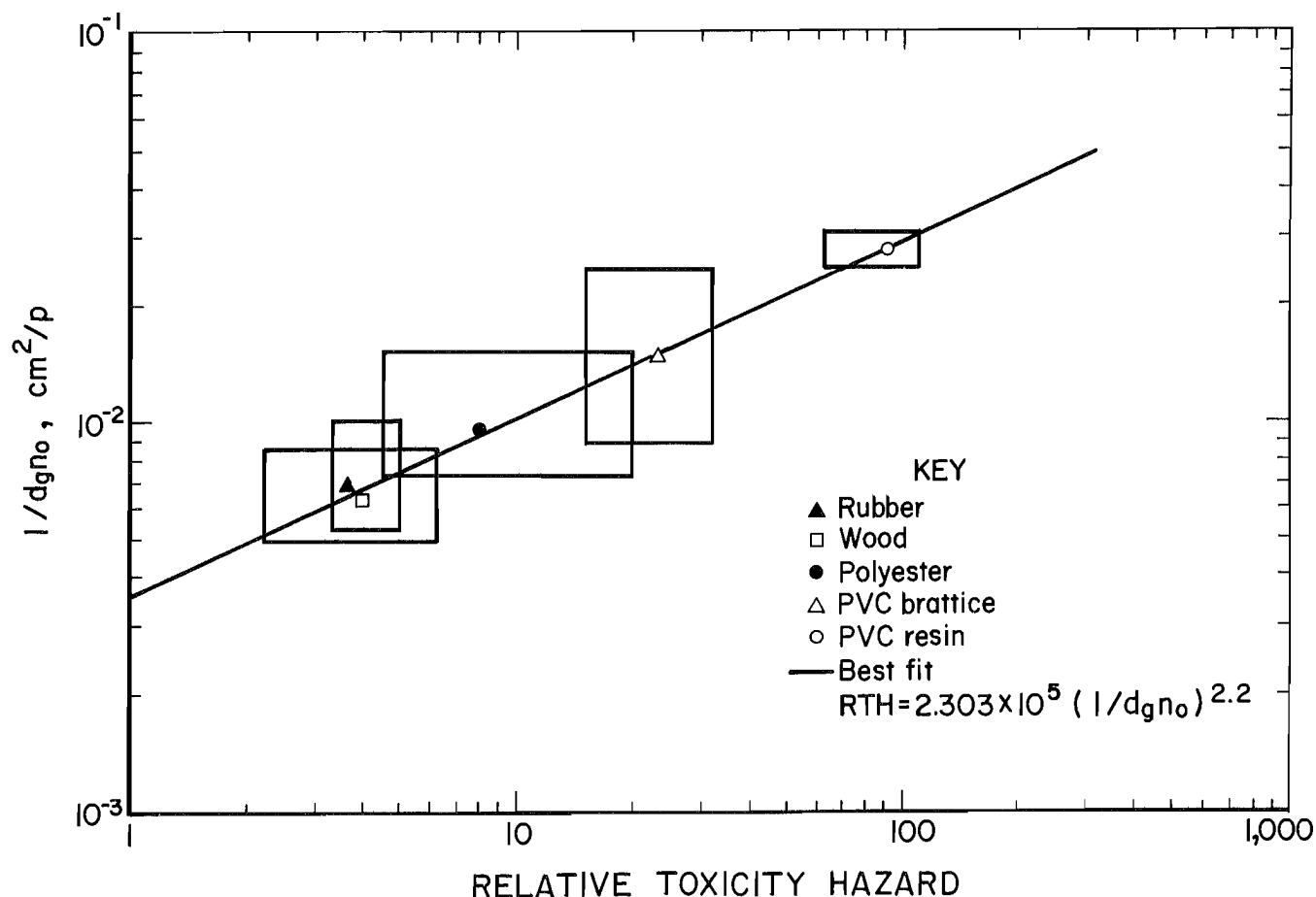


Figure 7.—Correlation of parameter $1/d_g n_o$ with toxic load data obtained by other researchers.

It could be argued that a low value of the parameter $1/d_g n_o$ (or corresponding high value of $d_g n_o$) represents some measure of the combustible's ability to generate toxic gas. At a high value of $d_g n_o$, a smaller percentage of the consumed mass is available for toxic gas production, while at lower values of $d_g n_o$, a higher percentage of the consumed mass is realized as toxic gas rather than smoke. Although this argument agrees qualitatively with the test results, no rigorous theoretical explanation is offered at this time.

Subsequent tests have been performed using mine conveyor belt samples with chlorine contents ranging from

1 to 20 pct. For these samples, the major toxic products were carbon monoxide (CO) and hydrogen chloride (HCl). The quantities of CO produced were relatively constant for all samples. It was found that the relative toxicity hazard parameter ($1/d_g n_o$), varied directly with the concentration of HCl produced, in excellent agreement with the above results.

The data acquired using the ionization chamber are sufficient to define both the relative smoke obscuration hazard and the relative toxicity hazard for combustible materials.

CONCLUSIONS

The results indicate that the use of an ionization chamber to measure the time-averaged value of the product $d_g n_o$, coupled with a simultaneous measurement of the sample mass loss Δm , provides sufficient information to determine both relative smoke obscuration and relative toxicity of combustible materials. Further, it was found that although different test conditions produce different values of the product $d_g n_o$ and mass loss Δm , the relative

values of the smoke obscuration and toxicity parameters represent reliable measures of the relative values of these hazards for different combustible materials. Therefore, these two parameters may be developed and used as test parameters to assess, simply and inexpensively, the toxic and smoke obscuration hazards of mine materials during fire.

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APPENDIX.–SYMBOLS

\bar{c}	root mean thermal ion speed, cm/s
C_o	normalization constant
d_g	average smoke particle diameter, μm or cm
$d_g n_o$	product of average particle diameter and concentration, p/cm^2
$1/d_g n_o$	relative toxicity hazard parameter, cm^2/p
$d_g^2 n_o$	relative optical density parameter, p/cm
$d_g n_o/\Delta m$	relative smoke obscuration hazard (RSH), $\text{p}/(\text{cm}^2 \cdot \text{g})$
D_i	positive ion diffusion coefficient, cm^2/s
d_p	individual smoke particle diameter, cm
e	electronic charge, esu (cgs)
e'	electronic charge, C (mks)
I_c	current produced by charged particles at third electrode, A
I_e	current measured in presence of smoke, A
k	Boltzmann constant, erg per kelvin
K	effective cross section for ionization chamber, $0.012 \text{ cm}^2/\text{p}$
\bar{K}	integrated value of Mie extinction coefficient
l	fixed separation
l_o	length of ionization chamber, cm
n^+	unipolar ion concentration, ions/cm^3
N_p	average charge of individual particle, number of positive ions
\bar{N}_p	average number of charges acquired by a system of polydisperse particles
n_o	smoke particle concentration, p/cm^3
Q_o	flow rate, cm^3/s
r	radius, cm
r_o	source electrode radius, cm
R_o	outer electrode radius, cm
RSH	relative smoke obscuration hazard
RTH	relative toxicity hazard
t	residence time of smoke particle in ionization chamber, s or min

T	temperature, kelvins
$T\ell$	transmission of light
TL	toxic load
V_c	voltage that amplifies current produced by charged particles at third electrode, V
V_o	voltage between the two electrodes, V
V_{oe}	voltage measured in presence of smoke, V
$V_{oe}(d_g n_o)$	voltage in presence of smoke, V
$(V_{oe})_o$	voltage in absence of smoke, V
γ	charging parameter
Δm	sample mass loss, g
η	ratio of voltage in presence of smoke to voltage in absence of smoke
μ^+	positive ion electrical mobility, $\text{cm}^2/\text{V} \cdot \text{s}$
σ_1	optical density